

## AMENDED SPECIFICATION

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# PATENT SPECIFICATION

911,204



NO DRAWINGS

911,204

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### COMPLETE SPECIFICATION

#### Bleaching Compositions

We, UNILEVER LIMITED, a Company registered under the laws of Great Britain, of Port Sunlight, in the County of Chester, England, do hereby declare the invention for 5 which we pray that a patent may be granted to us and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to bleaching compositions and in particular to detergent compositions capable of bleaching at relatively low temperatures.

Many detergent compositions contain a so-called persalt which provides a satisfactory bleach when the composition is used at or near the boil but which does not act rapidly enough at temperatures existing, for example, under normal conditions of use in unheated washing machines (50 to 70°C.) to provide a satisfactory bleach under these conditions. (These substances are believed to be not true persalts but to contain hydrogen peroxide of crystallisation which is liberated when the substances are dissolved in water. They are, therefore, more properly termed peroxyhydrates although they are hereafter referred to as persalts, as in the normal usage in the art. Examples of these compounds are the alkali-metal perborates, percarbonates, perpyrophosphates and persilicates.

The bleaching performance of persalts in detergent compositions at the relatively low temperatures prevailing in unheated washing machines can be improved by the inclusion of an agent enhancing the bleaching effect of hydrogen peroxide at low temperatures, here-

inafter called a low-temperature bleach enhancing agent.

By a persalt is meant a substance containing hydrogen peroxide of crystallisation which is liberated when the substance is dissolved in water.

By a "low-temperature bleach enhancing agent" is meant an agent not itself a bleach which, when incorporated in the detergent powder-sodium perborate composition employed in Section (2) of the following test, results in an improved degree of stain removal in comparison with the detergent powder-sodium perborate composition itself, when the washing procedure of Section (2) is followed.

#### TEST SECTION 1

White cotton sheeting to be employed in the test is first washed in a 0.5% solution of commercially available soapless detergent powder (built with phosphate and fluorescer) for 10 minutes in a washing machine. This ensures a good white background which assists in the assessment of faint stains. The cloth is rinsed, dried, ironed and divided into pieces approximately 60 cm x 70 cm.

The cloth is then stained with the following materials; Port wine, Burgundy wine, tea, Tomato soup, Oxtail soup, blackcurrant juice, tea with milk, tea with milk and sugar, coffee, blackcurrant jam, raspberry jelly, damson juice. Each piece carries a different stain. In all cases a teaspoonful of the staining material is applied. If there is a large proportion of solid matter (e.g. as in jams or certain

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types of soup) the excess is scraped off with a pallet knife. The stain should be about 15 cm. in diameter.

5 The staining materials are prepared as follows:

10 Port  
Burgundy  
blackcurrant juice  
blackcurrant jam  
raspberry jelly  
damson juice:

These are used as normally available.

Oxtail soup  
Tomato soup:

15 A tin of the soup is emptied into a saucepan with one third of a tin of water; the soup is stirred and warmed until the mixture is homogeneous.

Coffee:

20 Boiling water (170 ml.) is poured onto ground coffee (4 g.) and allowed to stand for 10 minutes and then strained through a muslin cloth.

Tea:

25 Boiling water (425 ml.) is poured onto tea leaves (12 g.) and allowed to stand 30 minutes and then strained through a muslin cloth.

Tea with milk:

30 80 ml. of the strained tea solution taken and 15 ml. fresh milk added.

Tea with milk and sugar:

80 ml. of the strained tea, 15 ml. fresh milk and 7.5 g. of sugar are stirred together.

35 After four days have elapsed the stained cloth is used in the test.

## SECTION 2

Performance of products containing bleaching agents in washing machines when used at concentrations of 0.01% available oxygen.

### 40 Product

- (i) Detergent powder similar to that given in Example 2 of British Patent No. 836,988 plus sodium perborate plus the agent to be tested.
- 45 (ii) Detergent powder similar to that given in Example 2 of British Patent No. 836,988 plus sodium perborate (control).

### 50 Washing Procedure

Eight gallons of suds containing 0.36% of

the detergent powder and an amount of sodium perborate sufficient to give a concentration of 0.01% available oxygen are prepared in a paddle-agitated washing machine at 60°C. One multi-stained test cloth together with 7 lb. of normally stained articles are immersed in the suds and agitated for 10 minutes. The test cloth is then removed, rinsed and dried.

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### Assessment of stain removal

The washed test cloths are examined 60 visually and each residual stain assessed on the following scale:

0 = stain removed  
 $\frac{1}{2}$  = stain visible with difficulty  
 1 = faint stain  
 2, 3, 4 = increasing degrees of residual stain.

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The totals of the stain scores for each test cloth are compared.

If the score given by Product (i) is lower than that given by the control, the added agent is considered to be a low-temperature bleach enhancing agent.

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The invention provides a detergent composition containing a persalt and a low-temperature bleach enhancing agent which detergent composition is in the form of a composite tablet wherein the low-temperature bleach enhancing agent is present in a section of the tablet from which is excluded any ingredient of the detergent composition detrimental to the stability of said agent, the latter substance being hereinafter referred to as separated material.

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The different sections of the composite tablet may be of any form and be associated together in any way to form a unitary tablet. Such a tablet may consist, for example, of a core, insert or layer or a number of cores, inserts or layers or a number of large granules or threads embedded in a matrix.

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It is immaterial in the present case in which section of the composite tablet the separated material is included, but as such material normally forms a relatively small proportion of a detergent composition it will normally be included in the core, insert or layer or similar section of the tablet and the active detergent, alkaline salts and the like of the detergent composition will form the matrix of the tablet.

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The dimensions of the section containing the separated material should be such as to secure effective stabilisation. In general, granules of diameter less than 0.25 cm. and threads of diameter less than 0.10 cm. should not be employed if the tablet consists of these embedded in a matrix.

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The section containing separated material may also be coated to increase stabilisation. Suitable coating materials are a paraffin wax or a solid fatty alcohol.

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It is also possible that the section containing

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the separated material may be in the form of a powder or granulate held within a closed cavity in the tablet and such a powder or granulate is included within the scope of the term insert.	65
5 It may be advantageous for the tablet to contain three or more distinct sections, one of which, for example, contains the low-temperature bleach enhancing agent, a second, the persalt, and a third the active detergent, alkaline salts and the like of the detergent composition.	70
10 It is normally desirable that the water content of the section containing a low-temperature bleach enhancing agent should be as low as possible but the water content of the other sections may generally be different and be governed by factors such as the ease of manufacture and the cohesivity and ease of disintegration of these sections.	75
15 The relative size of the different sections of the tablet is not critical. Although, as stated, the separated material will normally be a relatively small proportion of the total composition, the section containing it may also contain a major proportion of such of the other ingredients of the composition as are not detrimental to the stability of the separated material on storage, so that it may be the 20 largest section of the tablet. For convenience of manufacture, this is not generally preferred, however.	80
25 Examples of low-temperature bleach enhancing agents which may be used are: the reactive esters described in British patent No. 836,988, the reactive amides described in British patent No. 855,735, and the organic acid anhydrides described in British patent No. 549,015 and U.S. patent No. 2,362,401.	85
30 35 40 Suitable low-temperature bleach enhancing agents include sodium p-acetoxybenzene sulphonate, phthalic anhydride and acetylsalicylic acid.	90
45 A preferred persalt is sodium perborate. The core, insert or layer of the composite tablet of the invention is preferably in the form of a solid mass formed by compression. In this case, a suitable disintegrating agent such as dry starch is preferably included therein and lubricants normally employed in making tablets such as talc, mineral oil, hardened vegetable oil, boric acid, stearic acid, and soap powder, may also be included.	95
50 The detergent composition may be any 55 solid composition of a type customarily employed in the art in which the active ingredient is a soap, an anionic synthetic detergent or a non-ionic synthetic detergent or a mixture of these ingredients, and which includes auxiliary materials, of which the various alkali metal phosphates (e.g. tripolyphosphate and higher polyphosphates, hexametaphosphates, pyrophosphates and orthophosphates), alkali metal silicates, sulphates	100
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#### EXAMPLE 1

The following Example relates to a tablet in which a pellet of a low-temperature bleach enhancing agent is embedded in a detergent composition containing a persalt.

A granular detergent composition containing 88% of the following powder

		Parts	hydride was found after storage for two weeks under similar conditions).
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	Sodium dodecylbenzene sulphonate	4	
	Sodium toluene sulphonate	70	
5	Sodium tripolyphosphate	20	
	Sodium sulphate	4	
	Condensate of nonylphenol with 10 molecular proportions of ethylene oxide	5	
	Starch	1	
10	Talc		

and 12% of sodium perborate (10.4% available oxygen) was prepared. 6 g. of finely-divided phthalic anhydride (passing a mesh of aperture 251 microns), 0.30 g. of starch and 0.06 of talc were thoroughly mixed then compressed into a pellet of 2.5 cm. diameter. This pellet was centrally placed in the bottom of a 5.75 cm. diameter die which was then charged with 50 g. of the detergent powder/perborate mixture. The contents of the die were then compressed into the form of a tablet having a pellet of phthalic anhydride firmly embedded in one face.

In a composite tablet prepared according to the above method there was no detectable decomposition of the phthalic anhydride after storage for two weeks at 28°C. and 70% relative humidity. In a detergent tablet of similar weight prepared from the same ingredients but in which the phthalic anhydride was not in a separate section of the tablet (90—100% decomposition of the phthalic an-

EXAMPLE 2

The following Example relates to a tablet in which a pellet of a low-temperature bleach enhancing agent and a pellet of a persalt are embedded in the detergent composition.

A phthalic anhydride pellet of weight 6 g. similar to that described in Example 1 was placed centrally in the bottom of a 5.75 cm. die which was then charged with 45 g. of the spray-dried detergent powder described in Example 1. A perborate pellet of diameter 2.5 cm. made by compressing a mixture of 6 g. of sodium perborate (10.4% available oxygen) and 0.06 g. of talc, was placed centrally on the spray-dried detergent powder. The contents of the die were then compressed to give a composite tablet having a pellet of perborate embedded in one face and a pellet of phthalic anhydride embedded in the opposite face.

EXAMPLE 3

The following Example relates to a laminated composite tablet in which a layer of a low-temperature bleach-enhancing agent is pressed onto a detergent composition containing a persalt.

A layer of 6 g. of sodium p-acetoxybenzene sulphonate was added to 50 g. of the granular detergent composition given below and a tablet formed by compression.

65	<i>Detergent composition</i>	%
	Alkyl sulphate (64.7% active detergent)	23.1
	Sodium perborate	8.0
	Alkaline sodium silicate	6.0
	Water	12.0
70	Sodium carboxymethylcellulose	1.0
	Sodium tripolyphosphate	49.9

Negligible decomposition was found when tablets of the above composition were stored for one month at 28°C./70% relative humidity and 20°C./90% relative humidity in sealed wax-laminated cartons.

The following results were obtained when

detergent tablets of similar weight prepared from the same ingredients but in which the sodium p-acetoxybenzene sulphonate was not in a separate portion of the tablet were stored in similar sealed wax-laminated cartons for four weeks.

85	Storage condition	Sodium perborate de-composition %	Sodium p-acetoxybenzene sulphonate decomposi-tion %
	20°C./90% RH	0—20	30—50
	28°C./70% RH	10—20	50—60

EXAMPLE 4

90 This Example relates to a 'currant bun' type of tablet.

6 g. of pellets of sodium p-acetoxybenzene sulphonate of approximately 0.5 cm. diameter

and 0.3 cm. depth were mixed with 50 g. of the granulated detergent composition given in Example 3 and compressed to form a tablet. Negligible decomposition of the perborate and 10—20% decomposition of the sodium p-

5      acetoxybenzene sulphonate was found after storage of the tablets for four weeks at 20°C./90% relative humidity and 28°C./70% relative humidity, in sealed wax-laminated containers.

EXAMPLE 5  
10      This Example relates to a tablet in which threads of a low-temperature bleach enhancing agent are embedded in a detergent composition containing a persalt.

Threads of diameter approximately 0.15 cm. were made by extruding a 70% sodium p-acetoxybenzene sulphonate: 30% coconut ethanolamide mixture through a perforated plate. Tablets were made employing 6 g. of the sodium p-acetoxybenzene sulphonate/coconut ethanolamide thread and 50 g. of the detergent composition given in Example 3.

Storage tests in sealed wax-laminated containers for four weeks gave the following results:

Storage condition	Sodium perborate de-composition %	Sodium p-acetoxybenzene sulphonate the composition %
25      20°C./90% RH	0—10	10—15
28°C./70% RH	0—10	30—40

WHAT WE CLAIM IS:—

1. A detergent composition containing a persalt and a low-temperature bleach enhancing agent which detergent composition is in the form of a composite tablet wherein the low-temperature bleach enhancing agent is in a section of the tablet from which is excluded any ingredient of the detergent composition detrimental to the stability of said agent.
2. A detergent composition according to Claim 1 wherein the separated material is present in a core.
3. A detergent composition according to Claim 1 wherein the separated material is present in an insert.
4. A detergent composition according to Claim 1 wherein the separated material is present in a layer.
- 45      5. A detergent composition according to Claim 1 wherein the separated material is present in granules of diameter greater than 0.25 cm.
6. A detergent composition according to

Claim 1 wherein the separated material is present in threads of diameter greater than 0.10 cm.

7. A detergent composition according to any preceding claim wherein the section containing the separated material is coated.

8. A detergent composition according to Claim 7 wherein the coating material is paraffin wax or a solid fatty alcohol.

9. A detergent composition according to any preceding claim wherein the persalt is sodium perborate.

10. A detergent composition according to Claim 9 wherein the low-temperature bleach enhancing agent is sodium p-acetoxybenzene sulphonate.

11. A detergent composition in the form of a composite tablet substantially as hereinbefore described with particular reference to the Examples.

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